15

Energy Research Advisory Board
to the
United States Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585
(202) 586-5444

14 SEP 89 15. 14
R.L. GARWIN

September 8, 1989

Subject: Chairman's July 20 Request for Summaries of Past and Present Cold Fusion Research and August 9 Request for Tritium Production Results

The following have been received to date from the following individuals in response to the above requests:

L. N. Lewis, California Institute of Technology (response to August 9 request)

M. J. Rafelski, University of Arizona (response to August 9 request)

N. P.D. Bond, Brookhaven National Laboratory (response to August 9 request)

O. J.L. Straalsund, Battelle (response to August 9 request)

P. A. Narath, Sandia National Laboratories (response to August 9 request)

Q. W. Appleton, Oak Ridge National Laboratory (response to August 9 request)

Immediately behind this memorandum is a cumulative index.

Bill
William L. Woodard
Panel Secretary

Enclosures

090889. WLW

INDEX

A. Bo U.R. Sundqvist, Uppsala University (response to both requests)

B. W. Scheid, Giessen University (responses to both requests)

C. M.M. Broer, AT&T Bell Laboratories (response to August 9 request)

D. J. Eridon, Naval Research Laboratory (response to August 9 request)

E. Y.E. Kim, Purdue University (response to July 20 request)

F. A Schriesheim, Argonne National Laboratory (response to July 20 request)

G. M.A. Prelas, University of Missouri, Columbia

H. L.R. Greenwood, ANL (response to August 9 request)

I. F. Besenbacher, University of Aarbus (response to July 20 request)

J. N.J.C. Packham (for Bockris), Texas A&M (response to August 9 request)

K. J. Paquette, Atomic Energy of Canada, Ltd.

L. N. Lewis, California Institute of Technology (response to August 9 request)

M. J. Rafelski, University of Arizona (response to August 9 request)

N. P.D. Bond, Brookhaven National Laboratory (response to August 9 request)

O. J.L. Straalsund, Battelle (response to August 9 request)

P. A. Narath, Sandia National Laboratories (response to August 9 request)

Q. W. Appleton, Oak Ridge National Laboratory (response to August 9 request)

CALIFORNIA INSTITUTE OF TECHNOLOGY

DIVISION OF CHEMISTRY AND CHEMICAL ENGINEERING
THE CHEMICAL LABORATORIES

August 25, 1989

William Woodard
DOE ER-6 3F-061
Office of Energy Research
1000 Independence Avenue, SW
Washington, DC 20077-9381

Dear Dr. Woodard:

As requested by John Huizenga, Co-Chair of the ERAB panel on Cold Fusion, please find enclosed a summary of tritium measurements made on Pd/0.1 M LiOD-D_2O/Pt cells used in our investigations of room temperature electrochemical fusion. In addition, we address specific points requested below:

1. Source and specific activity of the D_2O used in the electrolytic cells: In all cases, we obtained 100 gm bottles of 99.5 (atom % D) D_2O from Aldrich Chemical Co. (Aldrich stock # 15,188-2). In the summary, D_2O A-D refer to measurements made on aliquots from four different bottles. The average ^3H specific activity from these four bottles was 148.0 ± 12.2 dpm cm^{-3} D_2O.

2. Description of the electrolytic cell: Electrolytic cells were made using 20 cm^3 glass vials fitted with polypropylene caps to prevent exchange of D_2O with ambient H_2O. Electrical leads were passed through holes cut in the caps, and the feedthroughs were sealed with epoxy resin to the caps. An additional small vent hole was made in the cap to allow escape of evolving gases. 0.1 M LiOD electrolyte was made by dilution of a stock solution of 1.0 M LiOD with 99.5 (%D) D_2O. The stock solution was made by the addition of Li wire to 99.5 (%D) D_2O. Pt foil anodes of *ca* 10 cm^2 area were

used in each cell. A loop of Cu/Sn wire (insulated except at the end) was passed through a hole punched in the Pt foil, crimped and epoxy sealed in place. Two Pd cathodes were used in the ^3H measurement cells. A 0.22 (diameter) x 10 cm cold worked Pd rod (first entry of the summary) was obtained from David Fell, Inc., Los Angeles. The rod was quoted at >99.9% Pd, although the actual manufacturing origin of this rod is unknown. The surface composition of the Pd rod was independently determined to be >99.9% by energy dispersive x-ray analysis. To remove any absorbed hydrogen, it was subjected to a 2 hr bake out at 300 °C in a 10^{-6} torr active vacuum. Another Pd rod, of 0.25 x 0.8 cm dimensions and >99.99% purity, was originally obtained as a 0.1 cm diameter piece from Johnson Mathey, Inc. The narrower wire was recast in an Al_2O_3 tube under 1 atm of CO(g) by melting the Pd at 1600 °C for 5 minutes and cooling at a rate of 50 °C/min through the melting point. The recast rod was then smoothed with sandpaper and ultrasonically cleaned in D_2O before use in the electrolytic cell. Electrical connections were made to both rods by crimping a loop of *ca* 0.5 mm diameter >99.99% Pt wire to the Pd. The Pd rods were placed in the center of a cell and each was surrounded by a Pt foil anode. Typically, each cell held about 15 cm^3 of 0.1 M LiOD/D_2O electrolyte.

3. Cell operating conditions: The 0.22 x 10 cm Pd electrode was anodized at 50 mA cm^{-2} for 20 minutes prior the beginning of the electrolysis experiment. The 0.25 x 0.8 cm Pd electrode was not subjected to any anodic activation before electrolysis. The electrolysis experiment was initiated upon passing a cathodic current (64 mA cm^{-2}, controlled current power supply) through the Pd electrodes. This current was maintained throughout the course of the experiment. In both cases, while bubble formation was evident at the anodes, no D_2 bubble formation was initially visible at either cathode. The potential that a typical Pd cathode adopted during electrolysis was measured in cells of comparable configuration *vs.* a Pd wire reference electrode charged with D to the α/β phase transition. Pd rods generally charged to -1.5 V *vs.* this reference potential at current densities of 64 mA cm^{-2}, and the IR corrected charging potential was $\approx$-0.8 V *vs.* the Pd/D reference. A charged specimen (0.025 cm x 2.3 cm wire, charged at 64 mA cm^{-2} for >12 hours) had an open circuit potential, as determined by current interrupt methods (5.0 ms after current interruption), of <-0.60 V *vs.* a Pd/D reference electrode. We

believe these to be representative values of the potentials that existed in the cells analyzed for tritium content.

4. Schedule of additions of D_2O during operation of the cell: At the current densities employed, we typically added 0.5-1 cm^3 day^{-1} (99.5% D) D_2O to the cell containing the 0.22 x 10 cm Pd cathode and a similar amount every 3-4 days to the 0.25 x 0.8 cm Pd cathode cell.

5. Tritium analysis: Three 1 cm^3 aliquots of electrolyte were removed from each cell at the time indicated in the summary table and analyzed for ^3H by liquid scintillation counting (LSC) methods. The LSC instrument used was a Beckman Instruments LS-5000TD scintillation counter. Each cell aliquot was placed in a plastic scintillation vial and neutralized to a pH of 6-7 by addition of 0.4 cm^3 of 0.4 M potassium hydrogen phthalate (KHP). 9 cm^3 of "Ready Gel" scintillation cocktail (a high ionic strength tolerant cocktail sold by Beckman Instruments) was added to the resulting analyte, the vial was capped and vigorously shaken for 30 s to effect mixing and then immediately counted. Counting times were 5 minutes in all cases. In converting the counts per minute to decays per minute (dpm), we have assumed the manufacturer's specified conversion efficiency of 40% for scintillation yield from true ^3H decays. No attempt was made to independently calibrate the cocktail efficiency. Blank counting experiments performed on empty vials, vials containing scintillation cocktail only, holding 1 cm^3 H_2O/ 9 cm^3 Ready Gel, and 1 cm^3 0.4 M KHP solution/ 9 cm^3 Ready Gel consistently gave a background of 45±5 dpm. In no case did we observe a statistically significant buildup of ^3H in our cells. The paranthetical notation (P/F) under the first entry in the summary table refers to the expected ^3H production rate for this cell employing the rate quoted in the original Pons/Fleischmann/Hawkins room temperature fusion paper. We note that ^3H scintillation experiments are subject to interference from chemiluminescent events. By not neutralizing 1 cm^3 aliquots of 0.1 M alkali (either H or D based), we could consistently collect from 10^3 to $>10^5$ dpm. Two such examples are included in the summary. We did not analyze head space gas or gases evolved from open circuited charged Pd rods for ^3H content.

6. Neutron production rate in each cell or in comparable cells: Neutron production rates for the two cells in the summary and in several comparable cells were measured with a sensitive (>10%

efficiency) neutron counter. The details of the counter and a complete summary of the experiments will be included with the full report of our experiments to the ERAB's panel on Cold Fusion. Nevertheless, from those experiments we may place an upper limit on the neutron production rate of *ca.* 200 η cm^{-3} (Pd) h^{-1} for the 0.22 x 10 cm Pd cell and *ca.* 1700 η cm^{-3} (Pd) h^{-1} for the 0.25 x 0.8 cm Pd cell.

7. Heat balance measurements: No calorimetric experiments were performed on the cells analyzed for ^{3}H. We have performed such heat balance experiments on comparable cells, and will make all the details available in the forthcoming full report to the ERAB. We note here, however, that we have not observed (to within ± 5%) excess heat in any of our calorimetric experiments.

8. Local tritium sources: Tritium analysis was performed in Prof. Judith Campbell's laboratory on the first floor of the Braun building. The science performed in the Braun building is essentially biochemical in nature; sources of ^{3}H exist throughout the building, including Professor Campbell's labs. We have recently checked with our campus health physics office and they report no incidents of ^{3}H contamination in this building. The cells analyzed for ^{3}H were set up on the second floor of Noyes and run in the sub-basement of Kellogg. Sources of ^{3}H exist in the basement and sub-basement of Noyes and in a separate laboratory at the opposite end of the sub-basement floor in Kellogg.

We hope this information is sufficient for the Panel's purposes. If there are any further questions, we would be happy to respond to them.

Sincerely,
Nate Lewis/(BJT)
Nate Lewis,
Associate Professor of Chemistry

Enclosure

cc: Prof. Jack Bigeleisen

Summary of ^3H Scintillation Experiments

Electrolyte	DPM[a]	Chemical Luminescence RCM[b], %	$^3H/cm^3\ D_2O$[c]	$^3H/cm^3$-Pd/s[d]
2.2 mm x 10 cm Pd **0.1 M LiOD/D_2O** **458 hours; 64 mA/cm^2** (KHP neutralized)[e]	**162.3 ± 12.7**	0.8 ± 0.1	**1.5×10^9**	5,435 (P/F: 127,300)
2.5 mm x 0.8 cm Pd **0.1 M LiOD/D_2O** **345 hours; 64 mA/cm^2** (KHP neutralized)[e]	**149.5 ± 12.2**	0.6 ± 0.1	**1.4×10^9**	20,600
0.1 M LiOD/D_2O (KHP neutralized)[e]	**144.0 ± 12.0**	0.5 ± 0.1	**1.3×10^9**	---
D_2O A	143.8 ± 12.0	0.6 ± 0.1	1.3×10^9	---
D_2O B	143.8 ± 12.0	0.5 ± 0.1	1.3×10^9	---
D_2O C	148.3 ± 12.2	0.4 ± 0.1	1.4×10^9	---
D_2O D	156.0 ± 12.5	0.3 ± 0.1	1.5×10^9	---
D_2O, average	**148.0 ± 12.2**	**0.4 ± 0.1**	**1.4×10^9**	**---**
0.1 M LiOD/D_2O	**29,970 ± 173**	**90.4 ± 0.7**	(2.8×10^{11})[f]	---
0.1 M LiOH/H_2O	**10,399 ± 102**	**90.2 ± 0.2**	(9.7×10^{10})[f]	---

a: Decays per minute for 1 mL analyte + 9 mL Ready Gel scintillation cocktail; assuming 40% efficiency for the scintillation fluor. Three determinations per entry. Values ± 1 standard deviation.

b: Random coincidence measure. Dpm perturbed by chemiluminescence above 5%.

c: Equivalent ^3H in 1 mL D_2O.

d: Rate of ^3H production calculated as difference from background (KHP neutralized 0.1 M LiOD/D_2O) per volume Pd for 20 ml total solution volume.

e: Neutralized by addition of 0.4 mL of 0.4 M potassium hydrogen phthalate, (KHP).

f: Apparent concentration ^3H; counts due almost exclusively to chemiluminescence.

THE UNIVERSITY OF ARIZONA
TUCSON, ARIZONA 85721 USA

COLLEGE OF ARTS AND SCIENCES
FACULTY OF SCIENCE
DEPARTMENT OF PHYSICS
BUILDING #81
(602) 621-6820

Dr. Ryszard Gajewski
US Dept of Energy/AEP

by FAX

August 31, 1989

Dear Ryszard:
Enclosed please find a preliminary note on our understanding of the question of tritium production in Cold Fusion.

It would be very kind of you if it would be possible for you to forward a copy of this note to ER-6, 3F-061 (William Woodard) as per their request of 9 August. As I have no FAX number for Dr. Bigeleisen, I also ask them to attend to any further distribution of this preliminary document, intended only for INTERNAL use by the committee.

Please limit distribution to official use only until submission for publication.

Yours Sincerely

Johann Rafelski
Professor of Physics

PRELIMINARY AZPH-TH/89-...

Comment on Tritium Production in Cold Fusion in Condensed Matter

David Harley and Johann Rafelski

Department of Physics
University of Arizona, Tucson, Arizona 85721

August 29, 1989

Following on the first report of the observation of cold fusion neutrons in laboratory, [1,2], Fleischmann *et al*[3] have also claimed that anomalous amounts of tritium may be produced in this way. While their other measurments of heat and million times higher neutron yield than that of Jones' have been repeatedly criticized, there have been recently several reports supporting the third claim that tritium is produced during the electrolysis of heavy water [4,5,6,7]. In particular, Packham *et al* have presented experimental data that places a lower limit on the tritium production rate in two such experiments with palladium cathode and nickel anode, vis. their runs labelled 'A2' and 'A7', for which we estimate a lower limit on the tritium production rate of $5.2 \times 10^6 \mathrm{ml}^{-1}$of electrolyte $\mathrm{s}^{-1}$and $2.1 \times 10^8 \mathrm{ml}^{-1}\mathrm{s}^{-1}$ respectively. The same group concurrently measured the neutron flux during the experimental runs. In one electrolytical cell they obtain a neutron count of about one per second [8] which is about 10^6 less than the expected value derived from conventional deuteron-deuteron (dd) fusion, assuming symmetry between the neutron and tritium producing branches of the dd-fusion reaction. While the neutron count rate is consistent with the observation of 2.45 MeV fusion neutrons reported first by SE Jones *et al*[2], the reported tritium yield requires some phenomenon other than the one associated with the results of Jones *et al.* According to Wolf [8] it is very unlikely for the observed tritium abundance to have been from an impurity within the electrodes used in the experiments.

There are two points presented here in view of which make the tritium production perhaps even more mysterious than the excess heat reported. Firstly, the non-observation of neutrons severely limits the final-state triton energy due to the possibility of a neutron producing deuterium-tritium (dt) secondary reaction. Secondly, the required smallness of this energy makes tritium production even less likely in an exit channel in which more than one charged particle is present (such as the proton-tritium (pt) exit channel of the dd reaction), due to the Coulomb suppression of such channels. These observations seem to imply that the only possible nuclear mechanism derives from some nuclear reaction *other* than dd, and we discuss all possible options without arriving at any satisfactory conclusion. Perhaps we should emphasize here that while we explore the consequences of energy and momentum conservation, which we presume to be sacred laws of physics, we are ignoring the fact that the lowest order quantum mechanical processes lead to rates for the processes we are discussing which are many orders of magnitude too small.

The constraint on the final state triton energy arises from the possibility of secondary fusion reactions being induced by high energy tritium produced in the 'cold fusion reaction'. It is well known that the dt reaction has an unusually large cross section that peaks at about 5 barn for a triton incident upon a deuterium target at an energy of about 70 KeV. The cross section is reduced to 2 barn at about 150 KeV, and falls off as $1/E_t$ at higher energies. If a high energy triton is produced in an environment containing substantial amounts of deuterium, it has a significant probability of undergoing a fusion reaction before being brought to rest. Specifically, the fraction of tritons of initial energy E that will fuse in a target is given by:

$$\frac{N_{fus}}{N_{inc}} \simeq \int_0^E dE' \frac{\sigma_{fus}(E')}{S(E')} \qquad (1)$$

$\sigma_{fus}(E)$ is the energy dependent fusion cross section. $S(E)$ is the stopping power of the target, and at low energies is generally dominated by electron ionization processes that dissipate the incident charged particle's momentum. We have computed this fraction in manner similar to our prior treatment of the suggested fractal fusion [9] as function of the incident triton energy, using experimental data for the fusion cross section for d+t→ α(2.8 MeV)+n(14.8 MeV) [10] and computing $S(E)$ for t incident upon a PdD target using a standardized approach [11]. Despite the relative smallness of the fusion fraction (7.9×10^{-12} fusions per triton at 10 KeV, 1.7×10^{-9} at 20 KeV, and increasing to 2.7×10^{-6} at 100 KeV) the absolute yield of secondary neutrons we should expect to see turns out to be forbiddingly high. For t-energies as small as 100 KeV we find that the secondary neutron production rate should be of the order of 14 neutrons ml^{-1}electrolyte s^{-1}(for run A2 of Packham), or 570 $ml^{-1}s^{-1}$(Run A7). For the less restrictive run (A7), only at a triton energy of 14 KeV does the secondary neutron flux begin to fall below the threshold of detectibility of neutrons (about $0.01s^{-1}$and assuming an electrolyte content of 10 ml). We assume in these estimates that all other neutron channels (such as from $d+d \rightarrow {}^3He+n$) are totally suppressed.

If tritium is indeed created with the required energy of less than about 20 KeV, then one can expect an asymmetry of greater than 50 : 1 favoring the unwanted neutron branch in the dd fusion reaction. This arises as a simple consequence of the appearance in the transition matrix element of not only the entrance channel wave function (containing the usual tunneling amplitude) but also the exit channel wave function, which in the case of the charged reaction products t+p being created at KeV energies will be suppressed due to the small amplitude of the Coulomb wave near to the nuclear channel radius in this exit channel. This effect is well known, such as in the Gamov description of α-radioactivity. Clearly, the ${}^3He+n$ channel does not suffer from this suppression, as there is no repulsive Coulomb force acting between the reaction products.

One may also wonder where the energy of fusion has gone, if tritons are formed at such small energies in the dd reaction. The only plausible answer is that such a reaction arises in association with the conversion of an electron, or a Bremsstrahlung photon. The phase space of the three final state particles favors equal momentum for each, but that implies that the momentum of the nuclei is only about 4 MeV/c. This corresponds to a very small energy for the triton, just a fraction of a KeV. While this is in accordance with the lack of secondary neutrons, such a tiny energy would lead to a strong branching into the neutron producing branch of the dd reaction because of the above- mentioned final state Coulomb effect. This observation is weakened to some degree by the possibility of (yet unexplained) long range neutron tunneling towards one of the deuterons, but one would expect such a process to produce energetic tritons.

Thus if indeed the tritium observed is due to cold fusion, we must find another nuclear reaction which will form tritons at just the "right" energy of about 20 KeV, higher energies being forbidden by absence of secondary neutrons, lower energies being made implausible by final state Coulomb suppression. We therefore turn now to consider all possible tritium producing, deuterium induced nuclear reactions, accessible in cold fusion. The number of direct exothermic (positive Q-value) nuclear reactions involving a deuterium reacting with a stable or sufficiently long lived nucleus with nucleon number (A), resulting in tritium and a nucleus (A−1), is indeed severely limited; in fact, a survey of all such exothermic reactions using available nuclear and atomic mass tables [12] reveals just fifteen such possibilities, and two other candidates with small endothermic reaction energy (negative Q-values) which could possibly change sign after reevaluation of the experimental data. The two exothermic reactions:

$d+{}^{183}W \rightarrow t+{}^{182}W$ (Q=46±4 KeV) and particularly
$d+{}^{201}Hg \rightarrow t+{}^{200}Hg$ (Q=19±6 KeV)

are the only two known reactions with a $Q-$value of less than 100 KeV. The other 13 reactions, aside from the usual dd case, are:

$d+{}^{6}Li$ (Q=2.557 MeV for the $\alpha+n+t$ channel);
$d+{}^{9}Be$ (Q=4.592 MeV);
$d+{}^{13}C$ (Q=1.311 MeV);
$d+{}^{17}O$ (Q=2.114 MeV);
$d+{}^{143}Nd$ (Q=133 KeV);
$d+{}^{145}Nd$ (Q=302 KeV);
$d+{}^{149}Sm$ (Q=385 KeV);
$d+{}^{179}Hf$ (Q=157 KeV);
$d+{}^{189}Os$ (Q=336 KeV);
$d+{}^{195}Pt$ (Q=152 KeV);
$d+{}^{235}U$ (Q=959 KeV);
$d+{}^{238}U$ (Q=105 KeV).

None of these 13 more energetic reactions can proceed via a small Q-value to an excited intermediate state in the daughter nucleus. The nuclear mass data currently available further shows that $d+{}^{163}Dy$ has an adopted Q-value of Q=−14±4 KeV, having been listed at earlier times as Q=5 KeV. Another case, $d+{}^{176}Lu$ has been listed with Q=66 KeV in older mass tables, but today the adopted value is Q=−36 KeV.

Thus if tritium is produced without the presence of a much larger neutron signature, we must conclude that a (d,t) reaction on a heavy nucleus has taken place by some unknown mechanism with the best candidate being the reaction involving ${}^{201}Hg$ (13.6natural relative abundance, resulting in a triton of 19±6 KeV, just barely within the tolerable limit for neutron production by secondary reactions. (Other such good candidates are ^{183}W (14.3% natural abundance) and perhaps also ^{163}Dy (24.9%).) In view of the obstacles to such a reaction and the preceding discussion we however conclude that the process of tritium production without accompanying neutron yield gives rise to even a greater scientific mystery as the purported anomalous heat effect.

Acknowledgement This research has been supported by the US Department of Energy, Division of Advanced Energy Projects.

References

[1] Jones, S.E. *Bulletin of the American Physical Society* **34**, 1228 (1989).

[2] Jones, S.E., Palmer, E.P., Czirr, J.B., Decker, D.L., Jensen, G.L., Thorne, J.M., Taylor, S.F. and Rafelski, J. *Nature* **338**, 737 (1989).

[3] Fleischmann, M., Pons, S. and M. Hawkins "Electrochemically Induced Nuclear Fusion of Deuterium", *J. Electroanal. Chem.* **261**, 301 (1989) and erratum.

[4] Packham, N.J.C., Wolf, K.L., Wass, J.C., Kainthla, R.C. and Bockris, J.O.M. "Production of Tritium from D_2) Electrolysis at a Palladium Cathode", Texas A&M preprint; K.L. Wolf, private communication.

[5] Storms, E.K., Talcott, C., public announcement of tritium observations at the level of 2×10^{12} tritons $ml^{-1}$of electrolyte, a level comparable to that of Packham [4].

[6] Sanchez, C., Sevilla, J., Escarpizo, B., Fernandez, F.J. and Canizares, J. "Nuclear Products Detection During Electrolysis of Heavy Water with Ti and Pt Electrodes" Universitad Autonoma de Madrid preprint, to appear in *Solid State Comm.*.

[7] Iyengar, P.K., "Cold Fusion Results from BARC Experiments", Presented at the Emerging Nuclear Energy Systems (ICENES V), Karlsruhe, Germany, to appear in Proceedings (World Scientific, Singapore).

[8] Wolf, K.L., Packham, N.J.C., Lawson, D.L., Shoemaker, J., Cheng, F. and Wass J.C. "Neutron Emission and the Tritium Content Associated with

Deuterium Loaded Palladium and Titanium Metals", presented at May 23-25 1989 cold fusion meeting in Santa Fe, New Mexico and preprint.

[9] Harley, D., Gajda, M. and Rafelski, J."Review of the Current Status of Cold Fusion", Arizona Preprint AZPH- TH/89-60, presented at the NATO Summer Institute on Nuclear Equations of State, Peniscola, Spain, May 28, 1989, to appear in proceedings , Plenum. Pub. Co, W. Greiner, editor; and Harley, D., Gajda, M. and Rafelski, J."Review of the Current Theoretical Status of Cold Fusion", Arizona Preprint AZPH-TH/89-61, presented at the ICENES V, Karlsruhe, W. Germany, to appear in proceedings, World Scientific, Singapore.

[10] Jarmie, N., Hardekopf, R. A., *Phys. Rev. C* **29**, 2031 (1984) and Los Alamos preprint.

[11] Ziegler, J.F., Biersack, J.P. and Littmark, U. "The Stopping and Ranges of Ions in Solids", Pergamon Press (1985).

[12] Wapstra, A.H. and Audi, G. *Nucl. Phys* **A432**, 1 (1985).

BROOKHAVEN NATIONAL LABORATORY

MEMORANDUM

DATE: September 1, 1989

TO: N. P. Samios

FROM: P. D. Bond pdb

SUBJECT: Tritium Production in Cold Fusion Cells

In response to the ERAB Panel request, we have not yet found any significant tritium production in Cold Fusion Cells. The specifics of the three efforts in these areas are given on the attached.

/rs
Attachments

Authors: R. Davis, J.R. Johnson, J.J. Reilly & R. Stoenner

Source and specific activity of D_2O used in the electrolytic cells.

This experiment was NOT done in an electrolytic cell but studies cyclic formation and decomposition of FeTiDx while monitoring the gas phase for tritium content.

Description of the electrolytic cell, including volume of D_2O.

N/A

Cell operating conditions.

N/A

Schedule of additions of D_2O during operation of the cell.

N/A

Tritium analyses:

a. Method of purification of samples for analysis: N/A
b. Analytical method: N/A
c. Schedule of cells examined for possible tritium production: N/A
d. Specific activity of D_2O,$D_2(g)$ and PhD_n as a function of time each cell: N/A

Neutron production rate in each cell or in comparable cells.

N/A

Heat balance measurements in each cell in which tritium is found or in comparable cells.

N/A

What tritium sources are located in the buildings where the electrolytic cells are operated and/or in the analysis stages.

N/A

Additional Information: There was no detectible buildup of tritium over the course of the experiment which extended from 5/31/89 to 8/14/89. However, the high initial tritium content of the D_2 precluded any determination of low level fusion rates. As the experiment is rather benign in terms of the expenditure of time and effort, it will be repeated with D2 having a very low tritium background level.

Authors: J. McBreen

Source and specific activity of D_2O used in the electrolytic cells.

The D_2O was obtained from the Isotopes & Special Materials Group of the Safeguards & Security Division at BNL. They gave the specific activity as 0.00060 µCi/ml.

Description of the electrolytic cell, including volume of D_2O.

The cell consisted of a Pd sheet cathode (20mm x 40 mm x 25µ), and a Pt screen anode. The electrolyte was 120 ml of 1 M LiOD. The cell also had a fuel cell electrode to recombine the gases. Only one cell was run.

Cell operating conditions.

The cell was operated in the sealed mode at a current of 1 A over a four month period. After four months, samples of the original electrolyte and the cell electrolyte were analysed for tritium by the S&EP Division of BNL.

Schedule of additions of D_2O during operation of the cell.

No additions of D_2O were made to the cell.

Tritium analyses:

a. Method of purification of samples for analysis: No purification was done.
b. Analytical method: N/A
c. Schedule of cells examined for possible tritium production: Only one cell was tested.
d. Specific activity of D_2O, $D_2(g)$ and PhD_n as a function of time each cell: N/A

Neutron production rate in each cell or in comparable cells.

N/A

Heat balance measurements in each cell in which tritium is found or in comparable cells.

N/A

What tritium sources are located in the buildings where the electrolytic cells are operated and/or in the analysis stages.

None. In any event the cell was sealed.

Authors: A. J. Davenport and H. S. Isaacs, BNL, et.al.

Source and specific activity of D_2O used in the electrolytic cells.

Brookhaven National Laboratory, Stock $3.6.10^{-4}$ μ curies/ml.

Description of the electrolytic cell, including volume of D_2O.

100 ml polyethytene bottle, Volume of D_2O=80ml

Cell operating conditions.

Constant current = 300mA

Schedule of additions of D_2O during operation of the cell.

Make-up for electrolysis losses

Tritium analyses:

a. Method of purification of samples for analysis: None
b. Analytical method: Luminescence
c. Schedule of cells examined for possible tritium production: None
d. Specific activity of D_2O,D_2(g) and PhD_n as a function of time each cell: None

Neutron production rate in each cell or in comparable cells.

No neutron production

Heat balance measurements in each cell in which tritium is found or in comparable cells.

Not measured

What tritium sources are located in the buildings where the electrolytic cells are operated and/or in the analysis stages.

None

Additional Information: No significant changes were observed in ^3H after testing for up to two (2) weeks.

Battelle

Pacific Northwest Laboratories
P.O. Box 999
Richland, Washington U.S.A. 99352
Telephone (509) 375-2250

Telex 15-2874
Facsimile (509) 375-2718

September 1, 1989

Dr. William Woodard
U.S. Department of Energy
ER-6, 3F-061
1000 Independence Avenue, SW
Washington, DC 20077-9381

Dear Dr. Woodward:

Subject: CLAIMS OF TRITIUM PRODUCTION IN ELECTROLYTIC CELLS

We have conducted no "cold fusion" experiments at PNL where we have knowingly produced tritium. We have, however, analyzed electrolytes from Texas A & M and the University of Utah which we are reporting to you by enclosing copies of the letters to those two institutions. Though we have described our procedure for analyzing the samples in our correspondence, the descriptions of the experiments should be prepared by those two institutions.

Currently we are participating in two activities that relate to "cold fusion:" 1) a closed-cell electrolysis experiment and, 2) a double-blind test on Pd electrodes from the Fleischmann-Pons laboratory in search for ^{4}He and ^{3}He. The electrolysis experiment is designed to determine whether excess heat can be generated by electrolyzing a 0.1 M ^{6}LiOD solution in a teflon cell fitted with a Pt recombiner, a 0.4 cm diameter x 5 cm length Pd cathode, and a Pt wire mesh anode (see attached schematic). Neutron and tritium production is being monitored as well as heat production. The cell is highly instrumented with a computerized data acquisition system that will allow us to calculate integrated heat balances. Though the experiment is underway, it is premature to discuss it further at this time.

The second activity is being conducted in cooperation with the University of Utah and several laboratories in the United States and Europe. The main purpose for the investigation is to determine if ^{4}He is being generated in heat-producing palladium electrodes from the Fleischmann-Pons laboratory. Though results are starting to come in from these laboratories, there remains the need to receive and collate the rest of the results, match them with a detailed history of the electrodes, write up the results, and go through a stringent peer-review process before results can be released. Though we intend to cooperate fully with the ERAB panel by reporting these results, they likely will not be available by the September 15 deadline.

Very truly yours,

J. L. Straalsund, Manager
Materials and Chemical Sciences Center

WRW/jeb

Attachments

cc: Professor Jacob Bigeleisen - State University of New York
John R. Huizenga - Co-Chairman, ERAB Panel on Cold Fusion
P. M. Pak - DOE-HQ
J. J. Sutey - DOE-HQ

Battelle
Pacific Northwest Laboratories
P.O. Box 999
Richland, Washington U.S.A. 99352
Telephone (509)

Telex 15-2874

June 6, 1989

Mr. Jeffrey C. Wass
Texas A&M University
Department of Chemistry
College Station, Texas 77843-3255

Dear Jeff:

Enclosed are the results of the tritium analyses of the samples you sent to us in mid-May. We were pleased with the inter-laboratory agreement in the tritium measurements. I hope we have the opportunity of collaborating further in the future. Please call me on (509) 376-5664 if you have any questions or comments concerning the data. Best wishes in your ongoing research.

Sincerely,

Dave

David E. Robertson
Group Leader, Nuclear Chemistry Section
Chemical Sciences Department

TRITIUM CONCENTRATIONS IN TEXAS A&M D2O SAMPLES

SAMPLE	SAMPLE TYPE	TRITIUM CONCENTRATION (dpm/ml)
J	D2O Blank	140 ± 88 [139 ± 4]*
I	0.1 M LiOD Blank	127 ± 87 [133 ± 4]
H	0.5 uCi Tritium Std	(8.08 ± 0.07)E5 [(7.97 ± 0.07)E5]
F	0.1 M LiOD from electrolysis cell	(1.96 ± 0.02)E6 [(2.06 ± 0.02)E6]**
G	Same as F, except from different cell	1170 ± 120 [1150 ± 20]

* Numbers in brackets were repeat analyses conducted 2 days later for longer counting times and larger sample aliquots for the low activity samples

** This sample was diluted with high-purity water and distilled prior to tritium analyses

Battelle
Pacific Northwest Laboratories
P.O. Box 999
Richland, Washington U.S.A. 99352
Telephone (509) 376-3777

Telex 15-2874
Facsimile (509) 375-2718

August 30, 1989

Professor Milton Wadsworth, Dean
Department of Metallurgy
University of Utah
Salt Lake City, Utah 84112

Dear Milt:

SUMMARY OF TRITIUM ANALYSES OF YOUR "COLD FUSION" ELECTROLYTE

As I discussed with you this morning, the ERAB Committee has requested that we supply them with a summary of our "cold fusion" activities, one of which was our tritium analyses of your samples. I appreciate your authorization for me to release our analyses to the Committee. Accordingly, I am summarizing in this letter the results that will be reported to them via a copy of this letter. I am sure they would appreciate a description of the experiments that produced the electrolytes that we have analyzed for tritium. I understand that you are preparing those results.

The most recent analyses, reported to you via Dr. Guruswami on July 21, are summarized below:

Sample #	[Net counts/min]	[atoms of tritium/ml] (x10^{-8})
D_2O BG 8100	0.36±0.45	1.1±1.5
D/M LOD F8600	0.22±0.45	0.7±1.5
Cell #6-1 16/25	1.84±0.46	5.6±1.5
Cell #6-1 #2	1.66±0.48	5.1±1.5
Cell #6-1 #3 6/24	2.13±0.48	6.5±1.5
Cell #4 6/30	1.36±0.48	4.2±1.5
Cell #5	1.02±0.46	3.2±1.5
Cell #6 6/30	1.02±0.46	3.2±1.5

All uncertainties are in terms of one standard deviation.

When I reviewed the results that we sent to you on June 1 with P. O. Jackson, who made the measurements in a liquid scintillating instrument (Beckman LS-8000), I discovered that the samples were added to a scintillating cocktail solvent (opti-fluor) as a minor volumetric constituent. By substituting the same quantity of ancient (low tritium content) water to the cells being counted, we obtained a background count-rate from cosmic rays, natural radioactive gamma rays from the surroundings and gamma emitters with our facility. That blank was subtracted from all measurements that include the sample before obtaining the counts per minute due to the sample. Thus, corrected results follow:

Sample #	[Net counts/min]	[atoms of tritium/ml] (x 10^{-8})
Blank (D_2O)	1.40±0.46	2.12±0.70
Blank (D_2O-Li)	0.87±0.47	1.32±0.71
Cell #3-1	6.20±0.51	9.40±0.81
Cell #3-5/25	6.50±0.52	9.85±0.82
Cell #6	3.00±0.48	4.55±0.74

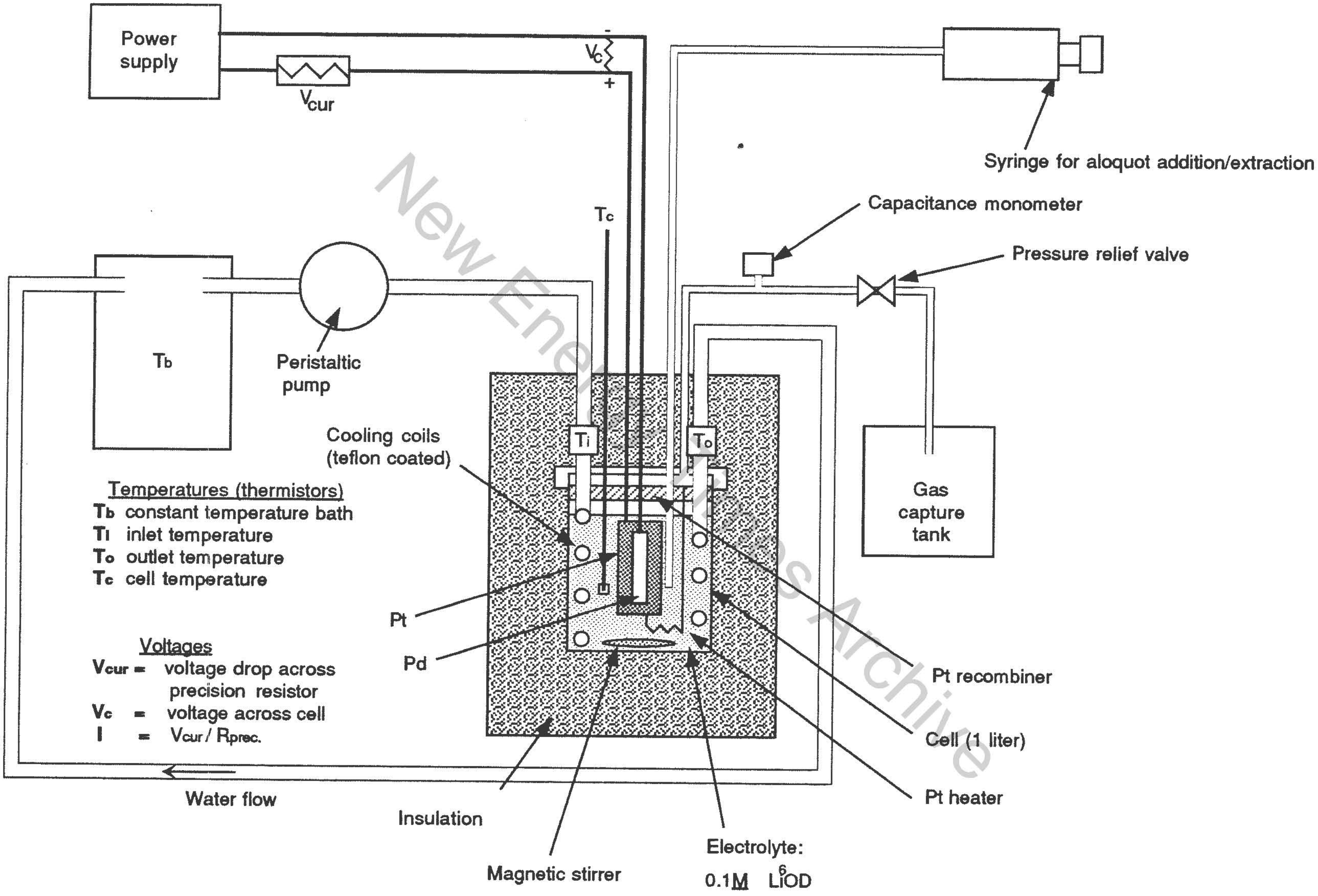
I mistakenly reported to you by phone that some of your samples had been distilled before they were counted. We did this with the first samples analyzed for Kevin Wolf at Texas A & M and found no significant difference in results counted before and after distillation. However, we have not distilled any of your samples. If important, chemiluminescence would have shown up in your D/M LOD F8600 and D_2O-Li samples. Quenching effects also appear not to be a problem in our method of analysis because our measurements on high-level, tritium-containing samples for Texas A & M show no appreciable differences before and after distillation, i.e., $1.8 \pm 0.2 \times 10^{13}$ and $2.0 \pm 0.2 \times 10^{13}$ atoms tritium/cc, respectively.

I am enclosing a summary report from Dr. Don Baer on the Auger electron spectroscopy, (AES), x-ray photoelectron spectroscopy (XPS), and secondary ion mass spectroscopy (SIMS) that we preformed on the section of electrode you supplied. I apologize for the delay in our report caused by the transfer of one of our staff members to the University of Maine.

Cordially yours,

John R. Morrey
Staff Scientist
Geochemistry Section

SCHEMATIC of D_2O EXPERIMENT



A. Narath
President

Sandia National Laboratories
Albuquerque, New Mexico 87185

AUG 28 1989

Dr. William Woodard
U.S. Department of Energy
ER-6, 3F-061
Office of Energy Research
1000 Independence Avenue, S.W.
Washington, DC 20077-9381

Dear Dr. Woodard:

Enclosed is the information requested by John Huizenga for the ERAB Panel on Cold Fusion. It concerns analyses for tritium production during cold fusion experiments.

I hope this information will be useful to the ERAB committee. If there are further questions please contact Tom Picraux at 505-846-7681 or 846-2009 (Fax).

Sincerely yours,

Enclosure

Copy to: Prof. Jacob Bigeleisen
State University of New York at Stony Brook

Copy to:

20	O. E. Jones
1000	V. Narayanamurti
1100	F. L. Vook
1110	S. T. Picraux
1840	R. E. Loehman
8340	W. Bauer
	Attn: K. Wilson, 8347

August 25, 1989

Tritium Production in Cold Fusion Cells at Sandia National Laboratories

We constructed a number of electrolytic cells to investigate cold fusion via electrochemical insertion of D into Pd or Ti cathodes. In these cells we used LiOD or LiOH, prepared from high purity (99.9%) naturally abundant lithium metal, as the electrolyte at 0.1 and 0.5 M concentrations made using 99.9% isotopic purity D_2O (Aldrich Chemical) or pyrolytically distilled H_2O. Table I details the cell configuration, operating conditions, experimental duration, and the resulting tritium content of the electrolytes after completion of the experiments. The tritium contents shown after completion of the experiments were not significantly different from contents measured during the course of the experiments except for the titanium tritide cell as discussed below.

In none of the cells detailed in Table I, nor in other electrolytic cells we operated, were the tritium contents or the neutron rates significantly above background. For tritium, background contents of cells using D_2O were 2-3 times as high as cells using H_2O. For the titanium tritide electrode, tritium content in the electrolyte was several orders of magnitude higher than the other experiments due to isotopic exchange of D in the electrolyte for T in the titanium matrix. The tritium content of this electrolyte increased with time.

As mentioned above, no electrolytic cells showed tritium contents higher than background at any time during the experiments. We were, however, able to find spurious, high tritium contents when cross-contamination was allowed between scintillation vials from Pd cells and scintillation vials from the titanium tritide cell. In particular, when a capped (not gas-tight) vial from a Pd cell was placed next to a capped vial from the titanium tritide cell for a period of 48 hours, the tritium content from the Pd cell was nearly 100 times that of the background. We prevented this cross-contamination by sampling and measuring the titanium tritide cell separately from the Pd cells. We also periodically checked the background in the analyzer and the laboratory (which contained 10 millicuries of tritium in a triple-sealed shipping cask) by measuring tritium contents of blank scintillation cocktails.

In one early electrochemical cell run with 0.1 M acetic acid the analysis of tritium was carried out for both the electrolyte and the Pd electrode. The conditions of this cell are listed in Table II. No tritium production was observed.

Table I. Summary of Electrochemical Fusion Cells[a]

Cell	Cathode	Electrolyte	Current Density (mA/cm²)	Cell Capacity (cc)	Duration (days)	Neutron Rate[e] (cpm)[f]	Tritium Content[g] (cpm)
1[b]	Pd rod, cast, annealed 6.3x0.32 cm	0.1 M LiOD	136-360	150	36	290	51.1
2[c]	Pd wire, extruded, annealed, 10x0.05 cm	0.1 M LiOD	65-990	100	61	100	49.1
3[c]	Pd rod, extruded, annealed, 4.6x0.32 cm	0.1 M LiOD	65-350	40	77	100	47.9
4[c]	Pd rod, extruded, annealed, 4.6x0.32 cm	0.1 M LiOH	65-350	40	77	100	16.9
5[d]	Ti tritide, thin film on Cu substrate, 6 cm²	0.1 M LiOD	50-300	100	5	1.2	$>10^6$

---

[a] Additional cells were operated under various conditions for shorter times, but are not detailed here.

[b] Cell used in freon vaporization calorimetry. D_2O makeup at 15-30 cc/day depending on applied current density. Calorimetrically measured heats were within 1 to 3% of the value predicted from the applied current and the thermoneutral potential for D_2O electrolysis indicating no excess heat generated by this cell.

[c] In addition to continuous operation at the current densities noted, cells 2, 3, and 4 were also pulsed between the low and high current densities to determine if neutron bursts occur. D_2O/H_2O makeup at 3-12 cc/day depending on applied current density.

[d] D_20 makeup at 5-20 cc/day depending on applied current density.

[e] Neutron rates reported using ^3He neutron detectors with efficiencies ranging from 1 to 4%. In no case were neutron counts significantly above background. Background counts are different for each cell due to cell location, shielding, and configuration of the neutron detector.

[f] Counts/min = cpm.

[g] Compared with background levels of 18.5 cpm for 0.1 M $LiOH/H_2O$ and 50.9 cpm for 0.1 M $LiOD/D_2O$. Tritium analyses performed on a Packard Model 1000 Tri-Carb Liquid Scintillation Analyzer using HiIonic Fluor Scintillation Cocktail. Other tritium sources were: (1) cell 1 was operated 70 meters and two floors from a hermetically sealed T source located in a connected building; (2) cells 2-5 were in a separate building containing no T sources; (3) the analysis system was located in a building which contained a T source in a hood 10 meters away and a small sealed standard used for calibration.

Table II. Combined Electrolyte and Electrode Analysis Cell

Cell[a]	Cathode	Electrolyte	Duration (days)	T in Electrolyte[b] (cpm)	T in Pd Rod[c] (cpm)	T Background[d] in D_2O (cpm)
1.1	Pd rod extruded, annealed, 2.56x0.32 cm	0.1 M CD_3COOD	14	483	412	468

---

[a] This cell was operated at a current density ranging from 40 to 200 mA/cm^2 and had a capacity of 40 mℓ. Only low sensitivity neutron monitoring was carried out and no excess counts above background levels (~1 cps) were observed.

[b] Analysis after two weeks. Similar results (478 cpm) were obtained after one week of operation.

[c] The Pd rod was dissolved in acid for tritium analysis.

[d] The D_2O of 99.75% purity was obtained from J. T. Baker Co. in 100 mℓ vials. The tritium background level varied for different batch numbers and in another case was 131 cpm.

Participants:

1111	J. A. Knapp
1821	M. J. Kelly
1821	H. D. T. Jones
1832	J. R. Scully
1832	F. S. Bovard
1841	S. S. Tsao
1841	T. M. Christensen
1841	T. R. Guilinger
1841	J. O. Stevenson
2147	N. A. Godshall
2512	D. Ingersoll
6224	E. P. Roth
6233	W. H. Casey
8343	D. F. Cowgill
8347	K. Wilson
9111	R. I. Ewing

OAK RIDGE NATIONAL LABORATORY
OPERATED BY MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE U. S. DEPARTMENT OF ENERGY
BILL R. APPLETON
ASSOCIATE DIRECTOR

POST OFFICE BOX 2008
OAK RIDGE, TENNESSEE 37831-6240
(615) 574-4321
FACSIMILE (615) 574-0323

September 1, 1989

Dr. William Woodard
U.S. Department of Energy
ER-6 3F-061
Office of Energy Research
1000 Independence Avenue, SW
Washington, DC 20077-9381

Dear Dr. Woodard:

The enclosed brief report summarizes the work at Oak Ridge National Laboratory related to possible tritium production in "cold fusion" experiments as requested in the ERAB memorandum dated August 9. Cold fusion experiments are being conducted by four separate groups at ORNL. Descriptions of each of these four efforts are enclosed. Only two of these groups have conducted experiments to date with a significant sensitivity to tritium production. The specific points raised in Huizenga's memorandum have been addressed for these two experiments. We will send further summaries of past and present cold fusion research at ORNL by the September 15 deadline. If you have questions or need further information please phone me.

Sincerely,

Barry L. Burks
for Bill R. Appleton

BRA:aj

cc: Jacob Bigeleisen

Internal Correspondence

MARTIN MARIETTA ENERGY SYSTEMS, INC.

August 29, 1989

B. R. Appleton

ERAB Request for Cold Fusion Reports

As per your request of August 22, the following information is included for claims of tritium production in "cold fusion."

The group in the Chemical Technology Division performed two experiments where stringent control of tritium inventory was carried out; one from May 31, 1989 to July 14, 1989 and another which was initiated on July 27, 1989 and is still in progress. The source of D_2O in both cases was Aldrich Chemical Company (low tritium level D_2O with 99.9 at. % D and an average tritium content of 1.7×10^3 Bq/L). Two other tests were made with D_2O that contained 10^3 more tritium and in which there was less attention to tritium levels.

Electrolytic Cells

The electrolytic cells were cylindrical in shape with active internal geometries of 4 cm diam x 10 cm height containing approximately 125 mL of electrolyte. For all but the most recent tests, an open system was used where the O_2 and D_2 were allowed to escape to the atmosphere and D_2O replenishment was performed approximately every 8 h. In our most recent test, a recombiner was placed in the space above the electrolyte in the form of a coil of Pt wire, coated with Pd-black. The electrolytically produced gases were recombined quantitatively to form D_2O, and no replenishment was needed except for the samples taken for tritium analysis (every other day). There was positive heat recovery by circulating cooling water.

The electrodes were Pd cathodes 0.28 to 0.55 cm diam x 8 cm and Pt wire anodes in the form of a coil that surrounded the cathodes. The electrolyte contained 0.1 to 0.2 N LiOD and operated in the temperature range of 25 to 33°C. The imposed electrical current density on the cathodes was 50 to 600 mA/cm^2.

Tritium Analysis

Tritium analysis was performed using a Hewlett Packard Tricarb 460C instrument. A 2-mL aliquot of the electrolyte sample was pipetted into 15 mL of the scintillation cocktail, and a standard and a blank sample were run for each analysis. No count rate dependence on LiOD concentration was found in the range 0 to 0.2 N LiOD.

The building in which the tests were performed has no other source of tritium.

Neutron Flux

The neutron flux was monitored continuously by a NE-213 scintillator with pulse shape discrimination and a threshold of 1.2 MeV that has a typical sensitivity at three standard deviations of 3 x 10^{-24} fusions/deuterium-pair.sec. A gamma spectrometer with a NaI crystal was used for detection of high-energy gamma rays.

Experimental Results

During the first two days of the test, identified as starting on May 31, 1989 utilizing an open system configuration, a tenfold increase in tritium concentration was observed in the electrolyte. This was an isolated incident which was not repeated and was not associated with power excesses observed in this experiment 500 h later (usually 5 to 10% excess energy for several hours). There was no apparent source of contamination. As the experiment proceeded, the amount of D_2O electrolyzed was replenished with fresh D_2O. This resulted in a gradual decrease in tritium concentration approaching that of the original D_2O.

A second experiment with the internal D_2O recombiner is still in progress. So far, no increase in the tritium concentration has been detected, but after 700 h of operation, there is an indication of a small power excess in the range of 3 to 5%.

Two other long-term experiments were performed with open systems utilizing D_2O with much higher initial tritium levels. There was no indication of significant increases in the tritium concentration, although there were relatively long periods of many hours in which there was apparent excess energy evolved in both tests.

C. D. Scott, 4505, MS-6226, ORNL (4-6775)

CDS:dw

cc: R. K. Genung
M. J. Saltmarsh

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: C. D. Scott

ORGANIZATION: Oak Ridge National Laboratory, Chemical Technology Division

SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

Electrochemical cells utilizing Pd cathodes and Pt anodes in 0.1 to 0.2 *N* LiOD were used. The electrolysis cell was insulated and a cooling jacket with circulating water used for heat removal. The neutron flux and gamma ray spectra were measured, and periodic electrolyte sampling for T was carried out. Electrical current densities of 100 to 600 mA/cm^2 Pd were used. In all completed experiments the cells were open, allowing the evolved D_2 and O_2 gasses to leave the cell. In the current, uncompleted experiment, an internal recombiner is being used so that the system is completely closed.

RESULTS AND COMMENTS:

In some of the open-system experiments, apparent excess power was detected for periods of several hours, usually in the range of 5 to 10%. However, during one 12-hour period an apparent imbalance of up to 50% was seen. These excursions, which were transitory, appeared after the current density was increased. They could be extended by perturbing the system, for example changing the electrolyte temperature or increasing the electrolyte concentration. There were two occasions, uncorrelated with any excess power observations, where the neutron detector count rate exceeded average background values by greater than three standard deviations. Recent, very preliminary results from a closed system, which includes a recombiner, are also indicating a power imbalance of ~5%, although this experiment is not yet complete.

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Pd	Pt wire
PURITY	99.9%	99.9+%
ALLOYING ELEMENTS	None	None
SOURCE OF MATERIAL	Materials Research Corp.	Englehard Corp.
PREPARATION CAST OR WROUGHT ANNEALED ATMOSPHERE VACUUM SPECIAL TREATMENT	Cast in Ar, swaged to appropriate diam, then annealed at 950°C under vacuum for 4 hours	
CHARACTERIZATION STRUCTURAL CHEMICAL BEFORE OR AFTER USE METHODS RESULTS	0.28-cm diam x 8-cm cylinder	24 ga wire
NOTABLE OBSERVATIONS	Periods of several hours of apparent excess power in the range of 5 to 10% were observed after changes to experimental conditions.	

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT __X__ yes ______ no
NEUTRONS ______ yes __X__ no
TRITIUM ______ yes __X__ no
HELIUM ______ yes __X__ no

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Pd, 0.28-cm diam x 9 cm	28 ga Pt wire
PURITY	99.9%	99.9+%
ALLOYING ELEMENTS	None	None
SOURCE OF MATERIAL	Materials Research Corp.	Englehard Corp.
PREPARATION **CAST OR WROUGHT** **ANNEALED** **ATMOSPHERE** **VACUUM** **SPECIAL TREATMENT**	Cast in Ar, swaged to appropriate diam	
CHARACTERIZATION **STRUCTURAL** **CHEMICAL** **BEFORE OR AFTER USE** **METHODS** **RESULTS**	0.28-cm diam x 9-cm cylinder	28 ga wire
NOTABLE OBSERVATIONS	Neutron count rate exceeded three standard deviations of the average background during a 4-hour period.	

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT ______ yes __X__ no

NEUTRONS ______ yes __X__ no

TRITIUM ______ yes __X__ no

HELIUM ______ yes __X__ no

ERAB Request for Cold Fusion Reports
Metals and Ceramics Division
Oak Ridge National Laboratory

E. L. Fuller, Jr. - Principal Investigator

The group in the Metals & Ceramics Division performed two types of experiments with sensitivity to tritium production. The source of D_20 in both cases was the Aldrich Chemical Company. Specific activity was measured to be 102 ± 6 disintegrations per minute (DPM) per milliliter. One experiment features calorimetry combined with tritium analysis; the other provides neutron counting with tritium analysis.

Electrolytic Cells

In one set of experiments a power calorimeter designed around a 40 gallon (water) temperature bath, controlled to 0.01°C or better, has been used with four cells in operation. Each cell holds 75 mL of electrolyte (either 0.1 N $^6LiOD/D_20$ or 0.1 N $^6Li\ OH/H_20$). Cells are brought on-line two at a time; one with light water and one with heavy water; so that the two cells are subjected to identical currents. No excess heat or tritium (above background) has been detected in these experiments to date. Further details may be found on the attachment.

In the other set of experiments six electrochemical cells using a variety of cathodes (different materials, sizes or treatments) were operated with BF_3 detectors for neutron counting. One cell did show several bursts of neutrons that lasted for 45 minutes, 60 minutes and up to 24 hours. No tritium (above background) has been detected in the these experiments to date. Further details may be found on the attachment.

In a separate experiment an electrolytic cell was operated without neutron counters and without calorimetric analysis that did show a burst of tritium activity in the electrolyte solution. This cell was constructed of yellow glass to exclude ultraviolet light from the reaction medium. A gold wire anode and a cast palladium rod cathode were operated in a volume of 150 to 200 milliliters. This cell was operated at currents ranging from 10 to 500 $milliamps/cm^2$. Replacement of depleted deuterium oxide was on a one or two day basis.

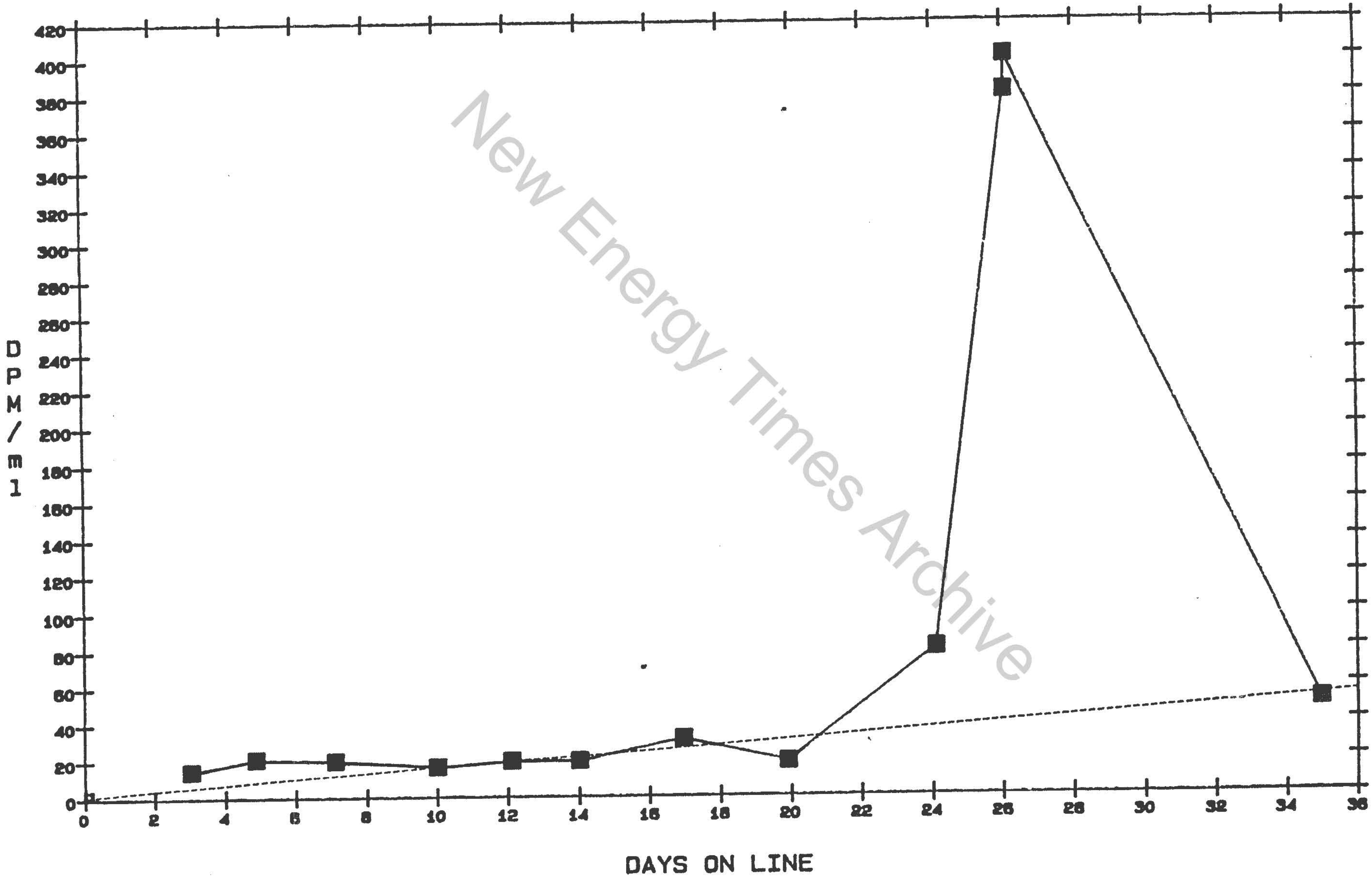
Experimental Results

Samples of electrolyte solution were taken at two to five day intervals for tritium analysis in a scintillation counter. The only other sources of tritium in the laboratory were tritium calibration sources stored in shielded containers. This cell did show a factor of ten enhanced tritium activity in one sample taken on the twenty-sixth day of operation. The analysis was repeated and the enhancement confirmed. The activity returned to background level for the next sample analyzed. This phenomenon has not been repeated since. A graph is attached of the tritium activity for this cell.

TRIT001GA

30-AUG-89 9: 32 Page 1

EXCESS TRITIUM: YELLOW GLASS CELL



MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: E. L. Fuller, Jr.

ORGANIZATION: Oak Ridge National Laboratory, Metals and Ceramics Division

SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

Six electrochemical cells using a variety of cathodes (different materials, sizes, or treatments) were operated in an arrangement suitable for neutron counting with BF_3 detectors. The cathodes for the six cells were as follows: (1) two cold-cast, swaged 3.1-mm-diam by 5-cm palladium rods, (2) one cold-cast 6.25-mm-diam by 5-cm palladium rod, (3) one 44-g cold-cast palladium "button," (4) one cold-cast 10.9-mm-diam by 3-cm palladium rod, and (5) one cold-cast 6.25-mm-diam by 5-cm titanium rod. The anodes were either platinum or platinum/rhodium wire and the electrolyte was 0.1 N $^{6}LiOD$. All cells were operated at current densities ranging from 25 to 300 mA/cm^2.

RESULTS AND COMMENTS:

Initially, a single BF_3 detector (~2% efficiency for Cf252 fission neutrons with ca. 200 counts per hour background), immersed in a water moderating bath, was used to monitor possible neutron emission from six surrounding electrochemical cells. After having observed an increase in the count rate of this detector, lasting about 18 hours and persisting even with the electrochemical cells removed from the bath, a second BF_3 detector was introduced to monitor the background level. Pulse height discrimination was used with both detectors to reduce noise counts, and a multichannel analyzer was used to observe the overall BF_3 output. Subsequently two unexplained increased in the BF_3 detector count rate were recorded while the experiment was unattended. In both of these instances the background monitor detector count rate remained constant. In one of these instances the count rate increased for about 24 hours. This incident was somewhat similar to the one that had led earlier to the introduction of the background monitor detector and a subsequent one in which the detector became very noisy for about 24 hours (the source of this noise was not discovered. In the second instance of increased count rate, two increases were recorded, lasting approximately 45 and 60 minutes, respectively, with the background monitor detector showing no corresponding increases. Such large increases in recorded count rates were occasionally observed in both counters and were caused by microphonics. Although the recorded increases in count rate while the experiment was unattended could have been caused by neutrons from the cells, not enough control existed in the experiment to conclude that this was definitely the case. The cell with a 44 gram electrode exploded (presumably $D_2 + O_2$) and since that date neither of the BF3 detectors have shown any variation above background (for about two months).

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Palladium, Titanium	Platinum or Platinum/Rhodium
PURITY	≥99.9%	
ALLOYING ELEMENTS	None	
SOURCE OF MATERIAL	In-house	
PREPARATION CAST OR WROUGHT ANNEALED ATMOSPHERE VACUUM SPECIAL TREATMENT	Cold-cast under Ar using copper molds. 3.1-mm rods were swaged from a single 6.25-mm rod.	
CHARACTERIZATION STRUCTURAL CHEMICAL BEFORE OR AFTER USE METHODS RESULTS	B-phase slowly undergoing a phase transition, but with no detectable α-phase (by x-ray diffraction). One electrode showed a large rhenium level after use (by glow-discharge mass spectrometry).	
NOTABLE OBSERVATIONS	The after-use palladium electrodes examined to date have shown a remarkable ability to retain deuterium. After several months, the D/Pd ratio in one electrode remained above 0.5.	
D/METAL RATIO ATTAINED	≥0.82 for Pd; not determined for Ti.	

EXPERIMENT YIELDED HEAT ______ yes __X__ no
NEUTRONS ______ yes ______ no (see text)
TRITIUM ______ yes __X__ no
HELIUM ______ yes __X__ no

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: E. L. Fuller, Jr.

ORGANIZATION: Oak Ridge National Laboratory, Metals and Ceramics Division

SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

A power calorimeter designed around a 40-gallon (water) temperature bath, controlled to 0.01°C or better, is currently in operation with four cells. Up to eight cells can be accommodated.

Each cell holds 75 mL of electrolyte (either 0.1 N $^6LiOD/D_20$ or 0.1 N $^6LiOH/H_2O$), and is equipped with a mechanical stirrer and calibration heater. The calorimeter cells can be operated over three power output ranges: (1) with an air jacket for very low power, (2) with a water jacket for intermediate power output, and (3) with no jacket (i.e., direct connection of the cell to the bath) for the highest power output.

RESULTS AND COMMENTS:

In this experiment, the cells are brought on-line two at a time; one with light water and one with heavy water. The current is connected in series between the two cells. This has the advantage of not only subjecting the cells to identical currents, but also providing a check on the electrolysis rate and the amount of makeup water required.

No excess heat or tritium (above background) has been detected in these experiments to date.

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Palladium	Ni Rod
PURITY	99.95%	
ALLOYING ELEMENTS	None	
SOURCE OF MATERIAL	Johnson-Matthey	
PREPARATION CAST OR WROUGHT	Wrought	
ANNEALED ATMOSPHERE VACUUM SPECIAL TREATMENT	Annealed at 600°C for 4 hours	Annealed at 600°C for 4 hours
CHARACTERIZATION STRUCTURAL CHEMICAL BEFORE OR AFTER USE METHODS RESULTS	In progress	
NOTABLE OBSERVATIONS	During the initial stages of electrolysis, a fine, black powder (probably nickel oxide) formed in the cell.	
D/METAL RATIO ATTAINED	Undetermined; experiment in progress.	

EXPERIMENT YIELDED HEAT ______ yes __X__ no
NEUTRONS ______ yes __X__ no
TRITIUM ______ yes __X__ no
HELIUM ______ yes __X__ no

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Palladium	Pt wire
PURITY	>99.9%	
ALLOYING ELEMENTS	None	
SOURCE OF MATERIAL	In-house	
PREPARATION CAST OR WROUGHT ANNEALED ATMOSPHERE VACUUM SPECIAL TREATMENT	Cold-cast under an Ar atmosphere in a copper mold	
CHARACTERIZATION STRUCTURAL CHEMICAL BEFORE OR AFTER USE METHODS RESULTS	In progress	
NOTABLE OBSERVATIONS	With the calorimeter design employed in this experiment, mechanical stirring is essential for obtaining a correct cell output power from the thermistor response (even at 200 mA/cm^2).	
D/METAL RATIO ATTAINED	Undetermined; experiment in progress.	

EXPERIMENT YIELDED HEAT ______ yes __X__ no
NEUTRONS ______ yes __X__ no
TRITIUM ______ yes __X__ no
HELIUM ______ yes __X__ no

TRITIUM MEASUREMENTS IN PHYSICS DIVISION
COLD FUSION EXPERIMENTS

The D_2O used in our cold fusion was obtained from the Y-12 plant at Oak Ridge. Our background measurements on this material yielded a tritium level of approximately 10^6 dpm/cc. Because of this high initial level and our emphasis on calorimetry, we made no attempts to measure tritium production on our initial experiments. We are now constructing four new experiments that will incorporate tritium measurements along with high accuracy calorimetry. For these experiments very low tritium (30 dpm/cc) heavy water has been obtained.

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: D. P. Hutchinson

ORGANIZATION: Oak Ridge National Laboratory, Physics Division

SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

Four calorimetry cells were operated for a period of over 1800 hours with two cells using 6.35-mm diam. x 10-cm long palladium cathodes in a 0.2 M ^{6}LiOD electrolyte, one cell with a similar cathode in a 0.1 M ^{6}LiOD electrolyte solution and one cell containing a cast 1.27-cm diam. x 10-cm palladium rod in a 0.2 M electrolyte. All cells were constructed with an electrolyte volume of 300 cm^3 and a 54 mm o.d. quartz envelope. No D_2/O_2 recombination was assumed. All of the cells contained a platinum wire spiral wound anode with a diameter of 30 mm. Two of the cells had 6.35 mm palladium rod electrodes precharged in D_2 gas to a stoichiometry of 0.6.

RESULTS AND COMMENTS:

The cells were operated at a current density of 50 mA/cm^2 for 48 hours and then at a current density of 250 mA/cm^2 for over 1800 hours. Three of the cells have remained in power balance within the experimental uncertainty of ±1 watt for the duration of the experiments. However, one of the cells, containing one of the non-precharged rods, exhibited an apparent power deficit of approximately 2 watts for the first 75 hours of operation, followed by an apparent power excess of approximately 3 watts for a 600-hour period beginning 150 hours after the beginning of the experiment. From 75 hours to 150 hours, the cell was in power balance. Shortly after the excess power was indicated, the cell was placed in a neutron counter containing a pair of NE213 scintillator detectors with pulse shape discrimination. The neutron emission level of the cell was determined to be less than 1 x 10^{-24} neutrons/s/D-D pair. Various changes were made to the cell during the period of apparent imbalance to improve the calorimetry measurement and to determine the effect of changes on the observation. The imbalance was not significantly affected by the bath temperature changes between 13 and 20°C, but disappeared with a reduction in temperature to 5°C. The apparent excess reappeared when the temperature was raised to 13°C, disappeared when the temperature of the bath was once again lowered to 5°C, and did not return after a temperature increase to 13°C. Subsequently, this cell has remained in power balance for 1100 hours. We are planning a thorough material analysis of the rods following the termination of these initial experiments in September 1989 and attempting to reproduce these observations.

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Palladium (6.35-mm diam x 10-cm long rod)	Platinum wire, #20 gauge
PURITY	99.96%	
ALLOYING ELEMENTS	None	
SOURCE OF MATERIAL	Johnson-Matthey	
PREPARATION CAST OR WROUGHT ANNEALED ATMOSPHERE VACUUM SPECIAL TREATMENT	Wrought Vacuum annealed @ 900°C for 2 hours	
CHARACTERIZATION STRUCTURAL CHEMICAL BEFORE OR AFTER USE METHODS RESULTS	In progress	
NOTABLE OBSERVATIONS	The cell that exhibited an apparent power imbalance was accidentally contaminated with a stainless steel screw in the electrolyte during assembly. It was not possible to remove the screw after the experiment began, and it remained in the cell for the entire duration. The significance of this is not known.	
D/METAL RATIO ATTAINED	As yet undetermined	

EXPERIMENT YIELDED HEAT ______ yes ______ no (See text)
NEUTRONS ______ yes __X__ no
TRITIUM ______ yes __X__ no
HELIUM ______ yes __X__ no

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: J. G. Blencoe

ORGANIZATION: Oak Ridge National Laboratory, Chemistry Division

SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

Investigation of reactions between high-purity palladium and deuterium at high gas pressures and low temperatures.

RESULTS AND COMMENTS:

A series of experiments were conducted to elucidate reactions between Pd and D_2 at high pressures and low temperatures. In our initial Pd-D_2 experiment, 0.7 grams of 0.1-mm thick palladium ribbon (Table 1) was wrapped around the tip of the lower (sample) thermocouple and sheathed with platinum foil. Pressurization of this sample to 380 MPa with 99.87% D_2 gas produced a rapid temperature rise (from 27 to 60°C) that was much larger than a simultaneous temperature rise (from 27 to 32°C) recorded by the reference thermocouple. This thermal anomaly decayed to the bath temperature after 12 minutes. During and for five days after this pressurization, the neutron flux was monitored at sampling times ranging from 6 seconds to 10 minutes. No sustained neutron flux above background was observed. Subsequent pressurizations of D_2 gas alone and 0.7 grams of palladium with H_2 gas produced thermal effects similar to the initial run, indicating that the thermal anomaly observed during our first Pd-D_2 experiment can be attributed to (1) PV work accompanying gas pressurization, (2) heat released during the formation of palladium deuteride, and (3) the somewhat different geometries of the sample and reference thermocouples.

The pressure vessel was then packed with 7.0 grams of palladium in the form of (1) ~1 gram of 0.1-mm thick ribbon wrapped around the sample thermocouple, and (2) ~6 grams of 3-mm x 3-mm cut pieces of 0.1-mm thick ribbon filling the space between the sample and reference thermocouples. Upon initial pressurization to 380 MPa with D_2 gas, thermal effects similar to those described above were observed. During the next three days, the temperature of the vessel was (1) reduced to -78°C using dry ice, and subsequently (2) allowed to warm slowly to room temperature. This was done because several investigators (e.g., Perminov et al., 1952) have reported greatly enhanced solubility of hydrogen in palladium at cryogenic temperatures. Finally, the sample was depressurized to a 20 micron vacuum at room temperature for one day and repressurized to 350 MPa for two days. As in the first Pd-D_2 experiment, no sustained neutron flux above background was observed.

I. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL	Palladium
PURITY	99.97 wt. %
ALLOYING ELEMENTS	See attached table.
SOURCE OF MATERIAL	Martin Marietta Energy Systems Stores, Y-12 Plant, Oak Ridge, Tennessee
PREPARATION CAST OR WROUGHT ANNEALED ATMOSPHERE VACUUM SPECIAL TREATMENT	See attached table.
CHARACTERIZATION	
STRUCTURAL	None.
CHEMICAL	See attached table.
BEFORE OR AFTER USE	Before.
METHODS	See attached table.
RESULTS	See attached table.
NOTABLE OBSERVATIONS	Deuterium continued to effuse from the palladium long after experimentation.
D/METAL RATIO ATTAINED	

EXPERIMENT YIELDED HEAT	X*	yes		no		don't know
NEUTRONS		yes	X	no		don't know
TRITIUM		yes		no	X	don't know
HELIUM		yes		no	X	don't know

*Heat released is believed to be entirely chemical (not nuclear).

Table 1. Analysis of 99.97% Palladium Ribbon[a,b]

Ag	6	As	0.7	Au	1	B	12	Ba	<0.2	Be	0.20	Bi	<0.1
Br	<0.04	Ca	73	Cd	<0.20	Co	0.02	Cs	<0.03	Cu	9	Fe	19
Ga	<0.03	Ge	<0.02	Hf	<0.05	Hg	<0.08	In	<0.01	Ir	3	K	0.50
Mg	0.70	Mo	2	Na	0.07	Nb	0.20	Ni	3	Os	<0.05	P	0.30
Pb	0.7	Pt	52	Rb	<0.01	Re	<0.02	Rh	29	Ru	<0.06	S	3
Sb	<0.05	Sc	0.02	Se	<0.01	Si	54	Sn	<0.30	Sr	0.03	Ta	< 5
Te	5	Th	0.06	Ti	2	Tl	<0.02	U	0.20	W	0.60	Y	<0.02
Zn	2	Zr	4	Cl	0.40	F	<0.01	I	<0.03	Ce	<0.10	Dy	<0.04
Er	<0.04	Eu	<0.02	Gd	<0.05	Ho	<0.01	La	<0.04	Lu	<0.01	Nd	<0.05
Pr	<0.01	Sm	<0.04	Tb	<0.01	Tm	<0.01	Yb	<0.04				

[a] Concentrations of all trace elements reported in ppm.

[b] Analyzed by DC-arc spectrograph at the Y-12 Plant, Oak Ridge, TN. Masked elements include Al, Cr, Mn, and V. Prior to use in experimentation, the ribbon was: cleaned in concentrated aqua regia; heated to 600°C under high vacuum (10^{-5} torr); exposed to 0.05 MPa D_2 gas at 600°C for 15 minutes; held at 600°C under high vacuum (10^{-5} torr) for one hour; cooled back to room temperature; and finally, stored under 99.999% argon. Additionally, to preclude surface contamination prior to experimentation, the ribbon was loaded into the pressure vessel under 99.999% argon.

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: J. G. Blencoe

ORGANIZATION: Oak Ridge National Laboratory, Chemistry Division

SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

Investigation of reactions between high-purity titanium and deuterium at high gas pressures and low temperatures.

RESULTS AND COMMENTS:

A single Ti-D_2 experiment has been conducted to test the claim that special disequilibrium pressure-temperature conditions will induce cold fusion in Ti-D_2 samples.

Starting materials for the Ti-D_2 experiment were (1) 1.6 mm-diameter titanium wire that was chemically polished in an acid bath prior to experimentation (Table 1), and (2) high-purity D_2 gas. The titanium wire (a total of 9 grams) was cut into 0.5- to 2.5-cm lengths before being subjected to high D_2 pressure. To prevent surface oxidation, the titanium wire was stored and transferred under 99.999% argon.

During the Ti-D_2 experiment, data were collected on pressure, temperature, and detector counts. In detail, the sequence of events was as follows: (1) pressurization to 380 MPa; (2) after approximately 29 hours (to compensate for a slow gas leak), repressurization to 380 MPa; (3) cooling to -78°C using dry ice, which produced a drop in pressure from 380 to 320 MPa; (4) slow warming to approximately 10°C over a 20-hour period, (5) depressurization to 80 MPa; (6) cooling to -196°C using liquid nitrogen, which produced a drop in pressure from 80 to 40 MPa; (7) slow warming to approximately -120°C over a 21-hour period, which produced an increase in pressure from 40 to 60 MPa; (8) rapid warming to 27°C over a 6-hour period, which produced an increase in pressure from 60 to 70 MPa; and finally (9) rapid cooling to -196°C, followed by rapid warming to 27°C, all within the space of an hour. After 105 hours of experimentation, the D_2 was vented from the pressure vessel and a vacuum pump was used to remove any residual gas.

The average detector count rate for the 105 hours of experimentation was 532.1 counts/hour. Approximately 80 hours after the experiment began, the count rate increased to ~590 counts/hour and remained at that level for about 5 hours. This increase cannot be due to random fluctuations in the average count rate and is potentially significant. If the increase can be attributed to neutrons emitted by the Ti-D_2 sample, this would correspond to an emission rate of ~1000 neutrons/hour.

However, because we cannot completely verify that our neutron detectors were operating properly during this 5-hour period, it cannot be claimed that cold fusion neutrons were observed. We are planning to repeat our Ti-D_2 experiment with an improved detector system where (1) moderated neutrons will be detected by two totally independent detector systems, (2) the background count rate will be monitored continuously by a similar and independent detector system placed nearby, and (3) additional shielding will be provided in an attempt to improve the neutron sensitivity of our detector systems.

I. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL	Titanium
PURITY	99.91 wt. %
ALLOYING ELEMENTS	See attached table.
SOURCE OF MATERIAL	Commercial source (See attached table.)
PREPARATION CAST OR WROUGHT ANNEALED ATMOSPHERE VACUUM	None.
SPECIAL TREATMENT	See attached table.
CHARACTERIZATION	
STRUCTURAL	None.
CHEMICAL	See attached table.
BEFORE OR AFTER USE	Before.
METHODS	See attached table.
RESULTS	See attached table.
NOTABLE OBSERVATIONS	No visible evidence of any reaction between the titanium and deuterium.
D/METAL RATIO ATTAINED	Not determined.

EXPERIMENT YIELDED HEAT	___ yes	X no	___ don't know
NEUTRONS	___ yes	X no	___ don't know
TRITIUM	___ yes	___ no	X don't know
HELIUM	___ yes	___ no	X don't know

Table 1. Analysis of 99.91% Titanium Wire[a,b]

C	35.0	N	15.0	O	500.0	Mg	<5.0	Si	<5.0	Al	20.0	S	10.0
Ca	5.0	V	5.0	Cr	40.0	Mn	30.0	Fe	150.0	Ni	40.0	Cu	30.0
Sn	10.0	Pb	20.0	Bi	<10.0								

[a] Concentrations of all trace elements reported in ppm.

[b] Lot analysis reported by vendor (#22/58107, Materials Research Corp., Orangeburg, NY). Prior to use in experimentation, the wire was chemically polished in an acid bath (nitric + lactic + hydrofluoric acid in a 5:5:4 ratio) to remove all surficial titanium oxide.